Microscale Zn Speciation in Smelter-contaminated Soils

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Introduction: To accurately speciate Zn in smelter-contaminated soils, we employed micro-XANES and micro-XRF spectroscopies. Surface (0-5 cm) and subsurface (5-25 cm) soil samples were collected, air-dried, embedded in an acrylic resin (LR-White resin), and polished into 50 Micron thin sections. Elemental maps were collected for Fe, Mn, and Zn over an area of 1 mm2 with 10 Micron steps. Using the generated map, XANES spectra were collected on "hot spots" (areas of highest metal concentration) 100 eV below the absorption edge to 300 eV beyond the absorption edge with increments varying from 0.05 to 10 eV. Spectra were collected on several Zn-bearing mineral phases and for Zn sorbed on reference oxides and clay minerals to aid in Zn speciation. In addition, samples were analyzed using bulk-XAFS and sequential extraction techniques in order to compare results using a variety of speciation methods. Micro-XAS and XRF results indicated that in the organicmatter rich surface soil the Zn was present as discrete Zn-sulfide (sphalerite) and Zn-Fe/Mn oxide (franklinite) mineral phases. This difference in Zn speciation in the surface soil occurred in an area of only a few hundred square microns, a difference not detected using standard XAS. In the subsurface soil, XAS revealed Zn to be predominately present as Zn sorbed to Fe and Mn oxides. This difference in Zn speciation in the surface and subsurface soils suggests weathering, transport and sorption processes have occurred. The results from this study show the utility of micro-focused spectroscopic studies relative to bulk-spectroscopic studies in determining contaminant speciation in soils.